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STRENGTH AND PLASTICITY

Analysis of the Structural State Formed in Titanium at the Final Severe Plastic Deformation Stage

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Abstract—The structural state of iodide titanium after deformation by drawing at 77 K, which is the final stage of severe plastic deformation (SPD), has been analyzed and estimated in this work. The SPD has been implemented by a sequential combination of deformation techniques (compression-squirting-extrusion-drawing) that provide different stress epures. The temperature dependence of the logarithmic damping decrement of torsional oscillations in the 77–250 K temperature range has been studied to give a physical interpretation of the nonmonotonic change in the strength of the iodide titanium after SPD and to compare the calculated strength with that obtained for low purity titanium.

Keywords: severe plastic deformation, drawing, internal friction, damping, strength, structural defects
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INTRODUCTION

Deformation-induced refinement is one of the main trends in the production of bulk metallic nanostructured materials which can be implemented using severe plastic deformation (SPD) [1]. Despite the existence of complicated SPD schemes (e.g., equal-channel angular pressing, isothermal forging, screw extrusion, and others [1–3]), we have shown for the first time that SPD, which can be used to create nanostructural materials, can be achieved by a sequential combination of deformation techniques that provide different stress epures (compression–squirting–extrusion–drawing) [4–9]. Deformation by drawing carried out at the final stage under cryogenic conditions (77 K) is an additional and important factor in structural refinement.

The aim of this work is to analyze and estimate the structural states of titanium that determine its mechanical properties depending on the deformation degree (drawing at 77 K). This dependence was previously revealed in [6] by the authors of this paper.

EXPERIMENTAL

After electron-beam remelting in a vacuum of 1.3×10^{-3} Pa, an ingot of iodide titanium was deformed according to the compression–extrusion–drawing scheme. Compression and extrusion were performed at 800K (true strain $e = 3.6$). The rod extruded 10 mm in diameter was then deformed by drawing to obtain a diameter of 3.75 mm ($e = 2$). The rod after the preliminary drawing was cut into two parts by the electric spark technique. One part of the rod was finally deformed by drawing in liquid nitrogen (77 K) and another was deformed at 300 K for comparison. The drawing under cryogenic conditions was carried out using special equipment described in detail in [10].

To analyze and estimate the structural state of titanium after the final stage of drawing, we measured the temperature dependence of the logarithmic damping decrement $\delta(T)$ of the torsional oscillations of low-frequency (0.5 Hz) internal friction in the 77–250 K temperature range using a reverse torsion pendulum in the heating mode with temperature stabilization (heating rate 0.025 K/s, measurement time of the damping decrement 150 s, vacuum 1.3×10^{-3} Pa) in the amplitude-independent region (strain amplitude 8×10^{-8}). Wire samples of at least 0.5 mm in diameter and 40 mm long were used. The measurement of $\delta(T)$ was performed on the samples after the final stage of SPD by drawing at 77 and 300 K at two true strains ($e_1 = 0.92$, $e_2 = 1.45$), corresponding to the extremum points of the microhardness-true strain dependences $H_\mu(e)$ calculated in [6]. Each experimental $\delta(T)$ curve is, as will be shown below, peak-shaped. This allowed each peak to be decomposed into components via computer calculation. The applied technique made it possible to calculate the activation energy of the processes and, consequently, to analyze the structural state of the titanium after different drawing stages. The

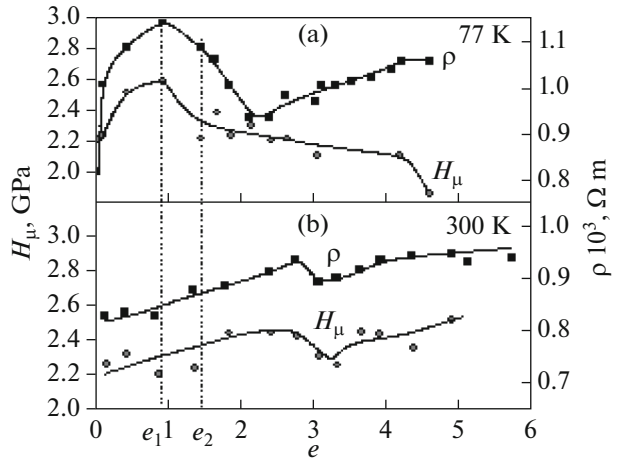

Fig. 1. Microhardness and electrical resistivity of titanium as a function of the true strain achieved by drawing at (a) 77 and (b) 300 K.

activation energy was calculated using the Wert–Marx formula [11]:

$$U = RT \ln\left(\frac{kT}{hf}\right), \quad (1)$$

where T is the temperature at which the internal friction maximum is observed, f is the measuring frequency, R is the gas constant, k is the Boltzmann constant, and h is the Planck constant.

RESULTS AND DISCUSSION

The investigation carried out in [6] showed that drawing at 77 K at the final stage of the SPD by compression–extrusion–drawing resulted in the nonmonotonic true-strain dependence of the microhardness and electrical resistivity (Fig. 1). At $e \approx 0.92$, there is a sharp increase in the microhardness and electrical resistivity, which indicates a significant increase in the concentration of deformation defects and, accordingly, an increase in the internal stress level. Further deformation by drawing at 77 K to $e > 0.92$ leads to a drop in the microhardness, which can be explained by internal stress relaxation due to the opening of microcracks formed from pores that have formed during vacancy coalescence. We should note that the probability of this process is high because vacancies form due to the intersection of boundary dislocations of unlike signs that are known to prevail over screw ones under cryogenic deformation conditions [12, 13]. It is noteworthy that the plastic deformation resistance level achieved by drawing at 77 K to $e = 0.92$, while maintaining the ductility of the iodide titanium, is the maximum possible and comparable to the strength of the low purity titanium [14, 15]. Analysis of the logarithmic damping decrement as a function of temperature that was measured in the present work, showed that cryogenic deformation up to e_1 and e_2 resulted in the wide peaks of the internal friction in the temperature range 100–175 K (Fig. 2). The deformation conditions have a considerable influence on the shape and height of the peaks. The internal friction peak after 77 K deformation to e_1 is substantially higher than that at e_2. This means that a higher concentration of linear and point defects is created in titanium at less true strain and maintained after storage at 300 K. Consequently, the defect concentration decreases rather than increases with increasing true strain from e_1 to e_2. It causes nonmonotonous change in the microhardness and resistivity after the final stage of SPD by drawing to various true strains. However, the fact that there is no noticeable difference in the width of internal friction peaks for both true strains under cryogenic conditions (Fig. 2a) indicates that the type (spectrum) of point defects that is determined by the energy of

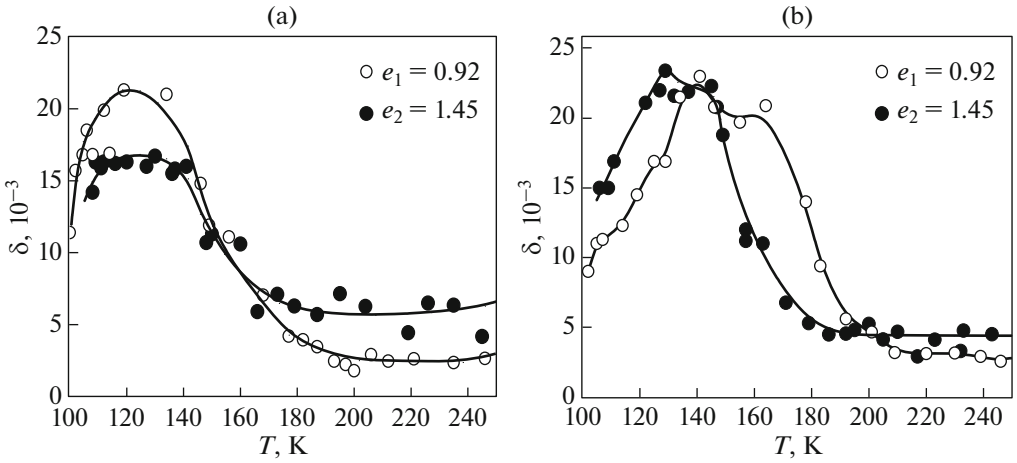

Fig. 2. Temperature dependencies of the internal friction of titanium deformed by drawing at (a) 77 and (b) 300 K to different true strains.

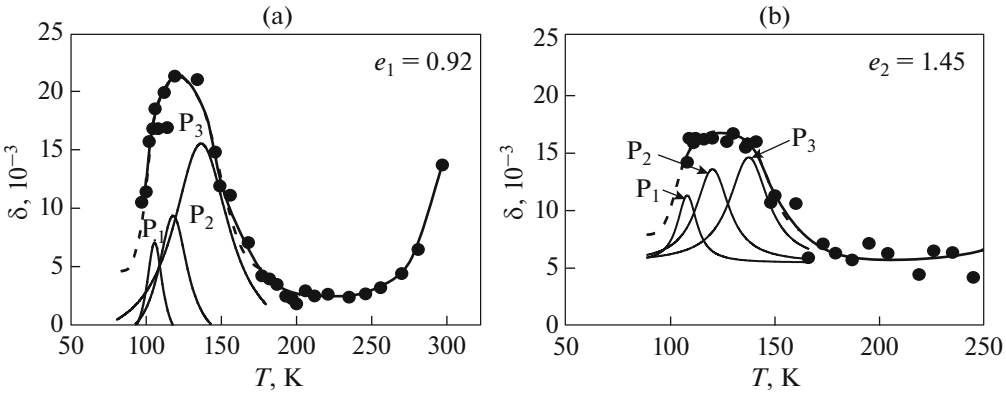

Fig. 3. Decomposition of the experimental temperature dependences of the internal friction in titanium into Debye peaks after drawing at 77 K to different true strains.

their bonding with dislocations, is independent of the true strain.

Let us analyze this assumption based on existing ideas about inelasticity phenomena in metals with a hcp lattice. Plastic deformation of such metals is known to change $\delta(T)$ in the temperature range 100–175 K and result in Hasiguti peaks that are caused by relaxation processes of interaction between linear and point defects (for example, the appearance and movement of double kinks on dislocations, as well as their interaction with point defects) [17, 18]. The deformation by drawing resulted in wide Hasiguti peaks and therefore these peaks cannot be described by the relaxation time only. This peak shape may indicate the presence of several overlapping peaks with the same relaxation time on the temperature scale. When analyzing Hasiguti peaks, they are usually decomposed into several Debye peaks [18, 19]. We found that each internal friction peak obtained after the decomposition of a wide peak is characterized by one activation energy that determines the interaction energy of a dislocation with different types of point defects. Three Debye internal friction peaks (Fig. 3) were formed as a result of the decomposition and the corresponding activation energies of each relaxation process were calculated. The results of the computer-aided calculation of the decomposition of the experimental $\delta(T)$ curves into the constituents and their corresponding activation energies estimated by Eq. (1) are listed in Table 1.

Analysis of the energy activation of the Debye peaks observed after the drawing suggests that the spectrum of point defects corresponds to the same

Table 1. Features of the Debye peaks of the internal friction in titanium deformed at 77 and 300 K to e_1 and e_2

Drawing temperature, K	True strain e	Temperature of the peaks after the decomposition of the experimental peaks, K	Activation energy of the process, eV	Type of point defects that interact with dislocations [16–18]
77	$e_1 = 0.92$	P_1, 104 P_2, 116 P_3, 136	0.27 0.31 0.36	Divacancies Monovacancies Vacancy–interstitial atom complex (H, O)
77	$e_2 = 1.45$	P_1, 110 P_2, 116 P_3, 137	0.29 0.31 0.37	Divacancies Monovacancies Vacancy–interstitial atom complex (H, O)
300	$e_1 = 0.92$	P_1, 114 P_2, 139 P_3, 166	0.30 0.37 0.44	Divacancies Vacancy–interstitial atom complex (H, O) Vacancy–interstitial atom complex (N, C)
300	$e_2 = 1.45$	P_1, 111 P_2, 127 P_3, 146	0.29 0.34 0.39	Divacancies Vacancy–interstitial atom complex (H, O) Vacancy–interstitial atom complex (O, N)

processes, namely, interaction between dislocations and divacancies, and vacancies and vacancy-interstitial atom complexes.

Importantly, the height of the Debye peaks after cryogenic deformation to e_1 is greater than that after deformation to e_2 (Fig. 3). Therefore, the concentration of linear and point defects of different types is greater after a smaller cryogenic deformation (e_1). This result explains the fact that the microhardness and electrical resistivity at e_1 are higher than those at e_2. It also confirms the previous assumption about the cause (due to the opening of microcracks or coalescence of vacancies in the monoatomic disks) of a sharp drop in the $H_\mu(e)$ and $\rho(e)$ dependencies after deformation to $e > e_1$.

A qualitatively different titanium structure forms during drawing at 300 K (Fig. 2b). In contrast to cryogenic deformation, the height of the internal friction peak after drawing at 300 K to e_2 is slightly higher than that after e_1. This means that the concentration of deformation defects increases with deformation, but insignificantly, which corresponds to a slight increase in the microhardness with increasing deformation (Fig. 1b). The experimental internal friction peaks that were obtained after deformation at 300 K, were similarly decomposed into Debye peaks and the corresponding activation energy of relaxation processes calculated for the interaction of dislocations with one type of point defects confirmed that processes with a higher activation energy of dislocation interaction with point defects develop at a lower true strain (e_1) than those at a higher true strain (e_2). However, the difference in these energies is not significant (see Table 1). This can result from the interaction between dislocations and vacancy-interstitial atom complexes of different impurities (H, O, N, C) with different atomic radii and, therefore, creating different levels of internal stresses. An internal friction peak at 166 K after 300 K deformation to e_1 that is missing after other deformation treatments, attracts attention. The activation energy (0.44 eV) indicates that this peak seems to be related to screw dislocations. An increase in the deformation to $e_2 = 1.45$ causes the peaks to shift towards low temperatures, i.e., the peak at 166 K with higher activation energy disappears. An analysis of the activation energy of the internal friction peaks suggests that the number of screw dislocations, edge dislocations, and edge dislocation-point defect complexes decreases significantly at $e_2 = 1.45$. Point defects responsible for the peak formation can be both deformation-generated vacancies and impurity atoms such as hydrogen [20]. Importantly, the height and the shape of the internal friction peak of titanium deformed at 300 K to $e_2 = 1.45$ coincides with those of titanium deformed at 77 K to $e_1 = 0.92$. The effect of increasing the deformation degree at 300 K on the peak shape is similar to that of decreasing the deformation temperature to 77 K. This fact is of practical importance because if the final sizes of the product is limited (e.g., wire diameter), the nanostructure state can be achieved even after small degrees of deformation by drawing, but under low-temperature (cryogenic) conditions.

CONCLUSIONS

The experimental temperature dependences of the low-frequency internal friction $\delta(T)$ for the iodide titanium allow us to draw the following conclusions.

(1) The maximum level of strength was achieved at the final stage of SPD by drawing at 77 K at small strain levels ($0.5 < e < 0.9$). The SPD was implemented by a sequential combination of the deformation techniques that provide different stress epures (compression-squirting-extrusion-drawing).

(2) An analysis of the energy activation of the interaction between point defects and dislocations suggests that the spectrum of point defects formed by drawing at 77 K to various true strains corresponds to the same processes, namely, interaction between dislocations and divacancies, and vacancies and vacancy-interstitial atom complexes.

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